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PATENT AND TRADEMARK OFFICE

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**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)**INTERNATIONAL APPLICATION NO.
PCT/GB00/00871INTERNATIONAL FILING DATE
March 9, 2000PRIORITY DATE CLAIMED
March 10, 1999TITLE OF INVENTION
POWDER COATING COMPOSITIONSAPPLICANT(S) FOR DO/EO/US
Andrew George CORDINER

Applicant herewith submits to the United States Designated/ Elected Office (DO/EO/US) the following items under 35 U.S.C. 371:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the international Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ have been transmitted by the International Bureaus.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 37(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). - **UNEXECUTED**
10. ☒ The International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

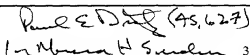
Items 11. to 16. below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

17. ☒ The U.S. National Fee (35 U.S.C. 371(c)(1)) and other fees as follows:

CLAIMS				
(1)FOR	(2)NUMBER FILED	(3)NUMBER EXTRA	(4)RATE	(5)CALCULATIONS
TOTAL CLAIMS	68 - 20	48	X \$ 18.00	\$ 864.00
INDEPENDENT CLAIMS	6 - 3	3	X \$ 80.00	240.00
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$ 270.00	<input type="checkbox"/>
BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)): CHECK ONE BOX ONLY				
<input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482)				\$ 690
<input type="checkbox"/> No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2))				\$ 710
<input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO				\$1000
<input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2) to (4)				\$ 100
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Surcharge of \$130.00 for furnishing the National fee or oath or declaration later than 20 30 mos. from the earliest claimed priority date (37 CFR 1.492(e)).				
TOTAL OF ABOVE CALCULATIONS				= 1,964.00
Reduction by 1/2 for filing by small entity, if applicable. Affidavit must be filed also. (Note 37 CFR 1.9, 1.27, 1.28).				= \$ 0.00
SUBTOTAL				= 1,964.00
Processing fee of \$130.00 for furnishing the English Translation later than 20 30 mos. from the earliest claimed priority date (37 CFR 1.492(f)).				+
TOTAL FEES ENCLOSED				\$ 1,964.00

- a. ☐ A check in the amount of \$__ to cover the above fees is enclosed.
- b. ☒ Please charge Deposit Account No. 16-1150 in the amount of \$ 1,964.00 to cover the above fees. A copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 16-1150. A copy of this sheet is enclosed.

18. ☐ Other instructions
n/a19. ☒ All correspondence for this application should be mailed to
PENNIE & EDMONDS LLP
1667 K STREET, N.W.
WASHINGTON, D.C. 2000620. ☒ All telephone inquiries should be made to (202) 496-4720Marcia H. Sundeen
NAME
SIGNATURE30,893
REGISTRATION NUMBER9-7-2001
DATE

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of: A. CORDINER

National Stage of PCT/GB00/00871

Group Art Unit: Unassigned

Filed: September 8, 2001

Examiner: Unassigned

For: POWDER COATING
COMPOSITIONS

Attorney Docket No.: 0156-024

PRELIMINARY AMENDMENT

Box PATENT APPLICATION

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Please enter the following amendments and remarks into the file of the above-identified application prior to the examination thereof.

IN THE ABSTRACT

Please add the following abstract:

---The invention provides a powder coating composition in which powder particles are an agglomerate of individual particulate components fused or bonded together into composite particles. Maximizing the amount of larger-sized uncoloured component(s) gives cost advantages, and the invention allows a rapid and flexible manufacture of a range of coloured powder coating compositions.--

IN THE CLAIMS

Please rewrite claims 1-68 as follows:

1. (Amended) A powder coating composition comprising composite powder particles that are an agglomerate of individual particulate components fused or bonded together, wherein the individual particulate components comprise

- (1) one or more colored film-forming base components having a Dv.99 of no more than 30 μ m, and
- (2) one or more uncolored film-forming components, wherein at least one of the uncolored film-forming components is an uncolored film-forming component (2a) that (i) has a Dv.99 that is more than 20 μ m and (ii) has a higher Dv.99 or higher Dv.50 than the one or more colored film-forming base components (1) taken together,

wherein the ratio of colored film-forming base components to uncolored film-forming components in the composition is in the range of from 1:99 to 60:40 by weight.

2. (Amended) The powder coating composition of claim 1, wherein the one or more uncolored film-forming components (2a) are present in an amount of at least 10% by weight of the total film-forming components (1) and (2).

3. (Amended) The powder coating composition of claim 1, wherein there is no more than 30% by weight, calculated on the weight of the total film-forming components (1) and (2), of uncolored film-forming components having a Dv.99 (i) that is no higher than the Dv.99 of the one or more colored film-forming components taken together, and (ii) that is less than 20 μ m.

4. (Amended) A powder coating composition comprising composite powder particles that are an agglomerate of individual particulate components fused or bonded together, wherein the individual particulate components comprise

- (1) one or more colored film-forming base components each having a Dv.99 of no more than 30 μ m, and
- (2)(a) one or more uncolored film-forming components each having a Dv.99 of more than 20 μ m and each having a higher Dv.99 and/or a higher Dv.50 than the one or more colored film-forming base components (1) taken together, and, optionally,

- (b) one or more other uncolored film-forming components.

wherein the one or more colored film-forming base components (1) is present in an amount of from 1 to 60% by weight of the total film-forming components (1) and (2), the one or more uncolored film-forming components (2a) are present in an amount of from 10 to 99% by weight of the total film-forming components (1) and (2), and the one or more other uncolored film-forming components (2b) are present in an amount of up to 30% by weight of the total film-forming components (1) and (2).

5. (Amended) The powder coating composition of claim 1, wherein there are two or more colored film-forming base components (1).

6. (Amended) A powder coating composition comprising composite powder particles that are an agglomerate of individual particulate components fused or bonded together, wherein the individual particulate components comprise

- (1) two or more colored film-forming base components having a Dv.99 of no more than 30 μ m, and
- (2) one or more uncolored film-forming components, wherein at least one uncolored film-forming components is a uncolored film-forming component (2a) that has a Dv.99 of more than 20 μ m and that is higher than the Dv.99 of the two or more colored film-forming base components (1) taken together,

wherein the ratio of the two or more colored film-forming base components (1) to the one or more uncolored film-forming components (2) is in the range of from 1:99 to 30:70 by weight.

7. (Amended) The powder coating composition of claim 6, wherein the uncolored film-forming components (2a) are present in an amount of at least 10% by weight of the total film-forming components (1) and (2).

8. (Amended) The powder coating composition of claim 6, wherein there is no more than 30% by weight, calculated on the weight of the total film-forming components (1) and (2), of uncolored film-forming component having a Dv.99 (i) that is no higher than the Dv.99 of the colored film-forming components taken together, and (ii) that is less than 20 μ m.

9. (Amended) The powder coating composition of claim 1, wherein there is at least one uncolored film-forming component (2a) that has a Dv.99 of at least 30 μ m.

10. (Amended) The powder coating composition of claim 9, wherein there is at least one uncolored film-forming component (2a) that has a Dv.99 of at least 35 μ m.

11. (Amended) The powder coating composition of claim 10, wherein there is at least one uncolored film-forming component (2a) that has a Dv.99 of at least 40 μ m.

12. (Amended) A powder coating composition comprising composite powder particles that are an agglomerate of individual particulate components fused or bonded together, wherein the individual particulate components comprise

- (1) two or more colored film-forming base components having a Dv.99 of no more than 30 μ m, and
- (2) one or more uncolored film-forming components, wherein at least one uncolored film-forming components is a uncolored film-forming component (2a) having a Dv.99 of more than 40 μ m,

wherein, the ratio of the two or more colored film-forming base components to the one or more uncolored film-forming components is in the range of from 1:99 to 60:40 by weight.

13. (Amended) The powder coating composition of claim 12, wherein the uncolored film-forming components (2a) are present in an amount of at least 10% by weight of the total film-forming components.

14. (Amended) The powder coating composition of claim 12, wherein there is no more than 30% by weight, calculated on the weight of the total film-forming components (1) and (2), of uncolored film-forming components having a Dv.99 (i) that is no higher than the Dv.99 of the colored film-forming components taken together, and (ii) that is less than 20 μ m.

15. (Amended) The powder coating composition of claim 1, wherein the ratio of colored film-forming base components (1) to uncolored film-forming components (2a) is in the range of from 1:99 to 50:50 by weight.

16. (Amended) The powder coating composition of claim 15, wherein the ratio of colored film-forming base components (1) to uncolored film-forming components (2a) is in the range of from 1:99 to 40:60 by weight.

17. (Amended) The powder coating composition of claim 16, wherein the ratio of colored film-forming base components (1) to film-forming components (2a) is in the range of from 1:99 to 30:70 by weight.

18. (Amended) The powder coating composition of claim 1, wherein the ratio of colored film-forming base components (1) to uncolored film-forming components (2a) is in the range of from 5:95 to 30:70.

19. (Amended) The powder coating composition of claim 1, wherein the uncolored film-forming components (2) are present in an amount of at least 50% by weight of the total film-forming components (1) and (2).

20. (Amended) The powder coating composition of claim 19, wherein the uncolored film-forming components (2) are present in an amount of at least 60% by weight of the total film-forming components (1) and (2).

21. (Amended) The powder coating composition of claim 20, wherein the one or more uncolored film-forming components (2) are present in an amount of at least 70% by weight of the total film-forming components (1) and (2).

22. (Amended) The powder coating composition of claims 2, wherein the one or more uncolored film-forming components (2a) are present in an amount of at least 20% by weight of the total film-forming components.

23. (Amended) The powder coating composition of claim 22, wherein the one or more uncolored film-forming components (2a) are present in an amount of at least 30% by weight of the total film-forming components.

24. (Amended) The powder coating composition of claim 23, wherein the one or more uncolored film-forming components (2a) are present in an amount of at least 40% by weight of the total film-forming components.

25. (Amended) The powder coating composition of claim 24, wherein the one or more uncolored film-forming components (2a) are present in an amount of at least 50% by weight of the total film-forming components.

26. (Amended) The powder coating composition of claim 25, wherein the one or more uncolored film-forming components (2a) are present in an amount of at least 60% by weight of the total film-forming components.

27. (Amended) The powder coating composition of claim 1, wherein there is no more than 15% by weight, calculated on the weight of total film-forming components (1) and (2), of the one or more uncolored film-forming components (2a).

28. (Amended) The powder coating composition of claims 1, wherein there are two or more compatible colored film-forming base components (1).

29. (Amended) The powder coating composition of claim 1, wherein the uncolored film-forming component (2a) or at least one of the uncolored film-forming components (2) is compatible with the colored film-forming base components (1) during film-formation.

30. (Amended) The powder coating composition of claims 1, wherein the uncolored film-forming component (2a) or at least one of the uncolored film-forming components is incompatible with the colored film-forming base components (1) or becomes incompatible with the colored film-forming base components during film-formation.

31. (Amended) The powder coating composition of claims 1, wherein the colored film-forming base components (1) contain a polyester.

32. (Amended) The powder coating composition of claim 30, wherein the uncolored film-forming component (2a) or at least one of the uncolored film-forming components (2) contains a polyester having a different functionality from the polyester of the colored film-forming base components (1).

33. (Amended) The powder coating composition of claim 31, which comprises an uncolored film-forming component (2) that contains a polyester, wherein uncolored film-forming component and the colored film-forming base components (1) contain different curing agents, wherein one of the curing agents is an epoxy curing agent or a co-reactable epoxy resin.

34. (Amended) The powder coating composition of claims 1, comprising an uncolored film-forming component (2) which is an agglomerate of an uncolored film-forming component fused or bonded to form composite particles with a non-film-forming component.

35. (Amended) The powder coating composition of claim 1, wherein uncolored film-forming component (2) includes a texturing additive.

36. (Amended) The powder coating composition of claim 34, which comprises a uncolored film-forming component (2) that includes mica.

37. (Amended) The powder coating composition of claims 1, wherein there is at least one uncolored film-forming component (2a) that has a Dv.99 of no more than 90 μ m.

38. (Amended) The powder coating composition of claim 1, wherein there is at least one uncolored film-forming component (2a) that has a Dv.90 of at least 14 μ m.

39. (Amended) The powder coating composition of claim 38, wherein there is at least one uncolored film-forming component (2a) that has a Dv.90 of at least 18 μ m.

40. (Amended) The powder coating composition of claims 1, wherein there is at least one uncolored film-forming component (2a) that has a Dv.90 of no more than 75 μ m.

41. (Amended) The powder coating composition of claims 1, wherein there is at least one uncolored film-forming component (2a) that has a Dv.99 in the range of from 50 to 65 μ m.

42. (Amended) The powder coating composition of claims 1, wherein there is at least one uncolored film-forming component (2a) that has a Dv.50 in the range of from 5 to 45 μ m.

43. (Amended) The powder coating composition of claim 42, wherein there is at least one uncolored film-forming component (2a) that has a Dv.50 of at least 8 μ m.

44. (Amended) The powder coating composition of claim 43, wherein there is at least one uncolored film-forming component (2a) that has a Dv.99 of at least 40 μ m and a Dv.50 of at least 10 μ m

45. (Amended) The powder coating composition of claim 43, wherein there is at least one uncolored film-forming component (2a) that has a Dv.50 in the range of from 12 to 30 μ m.

46. (Amended) The powder coating composition of claims 1, wherein at least one of the colored film-forming base components (1) has a Dv.99 in the range of from 6 to 25 μ m.

47. (Amended) The powder coating composition of claims 1, wherein at least one of the colored film-forming base components (1) has a Dv.50 of no more than 18 μ m.

48. (Amended) The powder coating composition of claim 47, wherein at least one of the colored film-forming base components (1) has a Dv.50 of no more than 15 μ m.

49. (Amended) The powder coating composition of claim 48, wherein at least one of the colored film-forming base components (1) has a Dv.50 in the range of from 2 to 12 μ m.

50. (Amended) The powder coating composition of claims 1, wherein the colored film-forming base components contain from 5 to 70 weight % of a pigment, calculated on the total weight of the colored film-forming base components.

51. (Amended) The powder coating composition of claims 1, wherein the colored film-forming base components contain from 0.5 to 50 weight % of the pigment, calculated on the total weight of the colored film-forming base components.

52. (Amended) The powder coating composition of claims 1, wherein the individual particulate components of the agglomerate include one or more non-film-forming components.

53. (Amended) The powder coating composition of claim 52, wherein the individual particulate components of the agglomerate include a texturing agent.

54. (Amended) The powder coating composition of claim 52, wherein the one or more non-film-forming components constitute up to 50% by weight of the particulate components of the composition.

55. (Amended) A kit for the preparation of powder coatings in a number of different colors comprising:

a plurality of differently colored film-forming base components, each with a Dv.99 of no more than 30 μ m, and

one or more uncolored film-forming components, each having a higher Dv.99 or a higher Dv.50 than the colored film-forming base components and each Dv.99 being more than 40 μ m.

56. (Amended) The kit of claim 55, wherein there is at least one uncolored film-forming components that has a Dv.99 of no more than 90 μ m.

57. (Amended) The kit of claim 55, comprising at least 7 differently colored film-forming base components.

58. (Amended) The kit of claim 55, comprising an uncolored film-forming component that is compatible during film-formation with the colored film-forming base components, and an uncolored film-forming component that is incompatible with the colored film-forming base components or that becomes incompatible with the colored film-forming base components during film-formation.

59. (Amended) A kit comprising for the preparation of powder coatings in a number of different colors comprising:

at least 7 differently colored film-forming base components,

an uncolored film-forming component that is compatible with the colored film-forming base components and remains compatible with the colored film-forming base components during film-formation and that has a Dv.99 of more than 40µm and no more than 90µm, and

an uncolored film-forming component that is incompatible with the colored film-forming base components or that becomes incompatible with the colored film-forming base components during film-formation and that has a Dv.99 of more than 40µm and no more than 90µm.

60. (Amended) The kit of claim 59, comprising means for comminution of the colored film-forming base components to a powder having Dv.99 of no more than 30µm.

61. (Amended) The kit of claim 55, wherein the colored film-forming base components contain a polyester.

62. (Amended) The kit of claim 55, comprising at least 3 uncolored film-forming components.

63. (Amended) The kit of claim 55, comprising means for agglomerating the components to produce a fluidisable powder.

64. (Amended) A process for preparing the powder coating composition of claim 1, which comprises providing the one or more colored film-forming base components (1) and the one or more uncolored film-forming components (2a) to provide a mixture of components and mixing and agglomerating the mixture of components into composite particles such that the composition is air-fluidisable and can be applied to a substrate by electrostatic spray.

65. (Amended) A process for preparing the powder coating composition of claim 1 from a kit comprising a plurality of differently colored film-forming base components and one or more uncolored film-forming components having a Dv.99 of more than 40µm, which comprises comminuting at least one of the colored film-forming base components to provide

a powder having a Dv.99 of no more than 30 μ m, and mixing and agglomerating the at least one comminuted colored film-forming base components and at least one uncolored film-forming component having a Dv.99 or a Dv.50 higher than the Dv.99 or Dv.50 of the at least one comminuted colored film-forming base components taken together, respectively, to form composite particles, such that the composite particles are air-fluidisable and can be applied to a substrate by electrostatic spray.

66. (Amended) The powder coating composition prepared by the process of claim 64.

67. (Amended) A process for forming a coating on a substrate, which comprises applying the powder coating composition of claims 1 to a substrate, and heating the applied composition to form a continuous coating.

68. (Amended) A substrate coated by a process of claim 67.

REMARKS

Claims 1-68, as amended, are pending in this application for the Examiner's review and consideration. Applicants have amended the claims to conform with U.S. patent practice and to more clearly recite the invention. As no new matter has been added herein, these changes should be entered.

Date

September 7, 2001

Respectfully submitted,

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Marcia H. Sundeen

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Appendix A

Changes to the Claims

1. (Amended) A powder coating composition comprising composite [in which] powder particles that are an agglomerate of individual particulate components fused or bonded together [into composite particles], wherein the individual particulate components comprise

- (1) one or more colored [coloured] film-forming base components having a Dv.99 of no more than 30µm, and
- (2) one or more uncolored [uncoloured] film-forming components, wherein at least one of the uncolored film-forming components is an uncolored film-forming component (2a) that (i) has a Dv.99 that is more than 20µm and (ii) has a higher Dv.99 or higher Dv.50 than the one or more colored film-forming base components (1) [or, when there is more than one coloured component, than those components] taken together,

wherein the ratio of colored [coloured] film-forming base components [component(s)] to uncolored [uncoloured] film-forming components [component(s)] in the composition is [being] in the range of from 1:99 to 60:40 by weight.

2. (Amended) The [A] powder coating composition of [as claimed in] claim 1, wherein the one or more uncolored [uncoloured] film-forming [component or] components (2a) are present in an amount of at least 10% by weight of the total film-forming components (1) and (2).

3. (Amended) The [A] powder coating composition of [as claimed in] claim 1 [or claim 2], wherein there is no more than 30% by weight, calculated on the weight of the total film-forming components (1) and (2), of uncolored [uncoloured] film-forming components [component(s)] having a Dv.99 (i) that is no higher than the Dv.99 of the one or more colored [coloured] film-forming [component or] components taken together, and (ii) that is less than 20µm.

4. (Amended) A powder coating composition comprising composite [in which] powder particles that are an agglomerate of individual particulate components fused or bonded together [into composite particles], wherein the individual particulate components comprise

- (1) one or more colored [coloured] film-forming base components each having a Dv.99 of no more than 30µm, [in an amount of from 1 to 60% by weight of the total film-forming components,] and
- (2)(a) one or more uncolored [uncoloured] film-forming components each having a Dv.99 of more than 20µm and each having a higher Dv.99 and/or a higher Dv.50 than the one or more colored [coloured] film-forming base components (1) [or, when there is more than one such component, than those components] taken together[, in an amount of from 10 to 99% by weight of the total film-forming components], and, optionally [if desired],
- (b) one or more other uncolored [uncoloured] film-forming components[, in an amount of up to 30% by weight of the total film-forming components].

wherein the one or more colored film-forming base components (1) is present in an amount of from 1 to 60% by weight of the total film-forming components (1) and (2), the one or more uncolored film-forming components (2a) are present in an amount of from 10 to 99% by weight of the total film-forming components (1) and (2), and the one or more uncolored film-forming components (2b) are present in an amount of up to 30% by weight of the total film-forming components (1) and (2).

5. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 4], wherein there are two or more colored [coloured] film-forming base components (1).

6. (Amended) A powder coating composition comprising composite [in which] powder particles that are an agglomerate of individual particulate components fused or bonded together [into composite particles], wherein the individual particulate components comprise

- (1) two or more colored [coloured] film-forming base components having a Dv.99 of no more than 30µm, and
- (2) one or more uncolored [uncoloured] film-forming components, wherein at least one uncolored film-forming component is an uncolored film-forming component (2a) that has [having] a Dv.99 of more than 20µm and that is higher than the Dv.99 of the two or more colored film-forming base components (1) [or, when there is more than one such component, of those components] taken together,

wherein the ratio of the two or more colored [coloured] film-forming base components (1) to the one or more uncolored [uncoloured] film-forming components [component(s)] (2) is [in the composition being] in the range of from 1:99 to 30:70 by weight.

7. (Amended) The [A] powder coating composition of [as claimed in] claim 6, wherein the uncolored [uncoloured] film-forming [component or] components (2a) are present in an amount of at least 10% by weight of the total film-forming components (1) and (2).

8. (Amended) The [A] powder coating composition of [as claimed in] claim 6 [or claim 7], wherein there is no more than 30% by weight, calculated on the weight of the total film-forming components (1) and (2), of uncolored [uncoloured] film-forming component[(s)] having a Dv.99 (i) that is no higher than the Dv.99 of the colored [coloured] film-forming [component or] components taken together, and (ii) that is less than 20µm.

9. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 8], wherein their is at least one [the] uncolored [uncoloured] film-forming

component [or at least one of the uncoloured film-forming components] (2a) that has a Dv.99 of at least 30µm.

10. (Amended) The [A] powder coating composition of [as claimed in] claim 9, wherein there is at least one [the] uncolored [uncoloured] film-forming component [or at least one of the uncoloured film-forming components] that has a Dv.99 of at least 35µm.

11. (Amended) The [A] powder coating composition of [as claimed in] claim 10, wherein there is at least one [the] uncolored [uncoloured] film-forming component [or at least one of the uncoloured film-forming components] that has a Dv.99 of at least 40µm.

12. (Amended) A powder coating composition comprising composite [in which] powder particles that are an agglomerate of individual particulate components fused or bonded together [into composite particles], wherein the individual particulate components comprise

- (1) two or more colored [coloured] film-forming base components having a Dv.99 of no more than 30µm, and
- (2) one or more uncolored [uncoloured] film-forming components, wherein at least one uncolored film-forming component is an uncolored film-forming component (2a) having a Dv.99 of more than 40µm,

wherein, the ratio of the two or more colored [coloured] film-forming base components to the one or more uncolored [uncoloured] film-forming components is [component(s) in the composition being] in the range of from 1:99 to 60:40 by weight.

13. (Amended) The [A] powder coating composition of [as claimed in] claim 12, wherein the uncolored [uncoloured] film-forming [component or] components (2a) are present in an amount of at least 10% by weight of the total film-forming components.

14. (Amended) The [A] powder coating composition of [as claimed in] claim 12 [or claim 13], wherein there is no more than 30% by weight, calculated on the weight of the total

film-forming components (1) and (2), of uncolored [uncoloured] film-forming components [component(s)] having a Dv.99 (i) that is no higher than the Dv.99 of the colored [coloured] film-forming [component or] components taken together, and (ii) that is less than 20µm.

15. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 14], wherein the ratio of colored [coloured] film-forming base components (1) to uncolored [uncoloured] film-forming components [component(s)] (2a) is in the range of from 1:99 to 50:50 by weight.

16. (Amended) The [A] powder coating composition of [as claimed in] claim 15, wherein the ratio of colored [coloured] film-forming base components (1) to uncolored [uncoloured] film-forming components [component(s)] (2a) is in the range of from 1:99 to 40:60 by weight.

17. (Amended) The [A] powder coating composition of [as claimed in] claim 16, wherein the ratio of colored [coloured] film-forming base components (1) to [uncoloured] film-forming components [component(s)] (2a) is in the range of from 1:99 to 30:70 by weight.

18. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 17], wherein the ratio of colored [coloured] film-forming base components (1) to uncolored [uncoloured] film-forming components [component(s)] (2a) is in the range of from 5:95 to 30:70.

19. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 18], wherein the uncolored [uncoloured] film-forming [component or] components (2) are present in an amount of at least 50% by weight of the total film-forming components (1) and (2).

20. (Amended) The [A] powder coating composition of [as claimed in] claim 19, wherein the

uncolored [uncoloured] film-forming [component or] components (2) are present in an amount of at least 60% by weight of the total film-forming components (1) and (2).

21. (Amended) The [A] powder coating composition of [as claimed in] claim 20, wherein the one or more uncolored [uncoloured] film-forming [component or] components (2) are present in an amount of at least 70% by weight of the total film-forming components (1) and (2).

22. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 2 [to 21], wherein the one or more uncolored [uncoloured] film-forming [component or] components (2a) are present in an amount of at least 20% by weight of the total film-forming components.

23. (Amended) The [A] powder coating composition of [as claimed in] claim 22, wherein the one or more uncolored [uncoloured] film-forming [component or] components (2a) are present in an amount of at least 30% by weight of the total film-forming components.

24. (Amended) The [A] powder coating composition of [as claimed in] claim 23, wherein the one or more uncolored [uncoloured] film-forming [component or] components (2a) are present in an amount of at least 40% by weight of the total film-forming components.

25. (Amended) The [A] powder coating composition of [as claimed in] claim 24, wherein the one or more uncolored [uncoloured] film-forming [component or] components (2a) are present in an amount of at least 50% by weight of the total film-forming components.

26. (Amended) The [A] powder coating composition of [as claimed in] claim 25, wherein the one or more uncolored [uncoloured] film-forming [component or] components (2a) are present in an amount of at least 60% by weight of the total film-forming components.

27. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 26], wherein there is no more than 15% by weight, calculated on the weight of

total film-forming components (1) and (2), of the one or more uncolored [uncoloured] film-forming components [component(s)] (2a).

28. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 24], wherein there are two or more compatible colored [coloured] film-forming base components (1).

29. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 28], wherein the uncolored [uncoloured] film-forming component (2a) or at least one of the uncolored [uncoloured] film-forming components [listed under] (2[a]) and (2b)) is compatible with the colored [coloured] film-forming base components (1) during film-formation.

30. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 29], wherein the uncolored [uncoloured] film-forming component (2a) or at least one of the uncolored [uncoloured] film-forming components [listed under] (2[a]) and [(2b)] is incompatible with the colored [coloured] film-forming base components [component(s)] (1) or becomes incompatible with the colored film-forming base components [therewith] during film-formation.

31. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 30], wherein the colored [coloured] film-forming base components [component(s)] (1) contain a polyester.

32. (Amended) The [A] powder coating composition of [as claimed in] claim 30 [and claim 31], wherein the uncolored [uncoloured] film-forming component (2a) or at least one of the uncolored [uncoloured] film-forming components [listed under] (2[a]) and [(2b)] contains a polyester having a different functionality from the polyester of the colored [coloured] film-forming base components [component(s)] (1).

33. (Amended) The [A] powder coating composition of [as claimed in] claim 31 [or claim 32], which comprises [includes] an uncolored [uncoloured] film-forming component (2) [(2a) or (2b)] that contains [containing] a polyester, wherein uncolored film-forming [that] component and the colored [coloured] film-forming base components [component(s)] (1) contain[ing] different curing agents, wherein one of the curing agents is [those being] an epoxy curing agent or a co-reactable epoxy resin.

34. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 33], comprising [which includes] an uncolored [uncoloured] film-forming component (2) [(2a) or (2b)] which is [itself] an agglomerate of an uncolored [uncoloured] film-forming component fused or bonded to form composite particles with a non-film-forming component.

35. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 34], wherein uncolored film-forming component (2) [(2a) or (2b)] includes a texturing additive.

36. (Amended) The [A] powder coating composition of [as claimed in] claim 34, which comprises an [wherein the] uncolored film-forming [agglomerated] component (2) that [(2a) or (2b)] includes mica.

37. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 36], wherein there is [the uncoloured film-forming component (2a) or] at least one [of the] uncolored [uncoloured] film-forming component[s] (2a) [or (2b)] that has a Dv.99 of no more than 90µm.

38. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 37], wherein there is [the uncoloured film-forming component or] at least one [of the] uncolored [uncoloured] film-forming component[s] (2a) that has a Dv.90 of at least 14µm.

39. (Amended) The [A] powder coating composition of [as claimed in] claim 38, wherein there is [the uncoloured film-forming component or] at least one [of the] uncoloured [uncoloured] film-forming component[s] (2a) that has a Dv.90 of at least 18µm.

40. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 39], wherein there is [the uncoloured film-forming component or] at least one [of the] uncoloured [uncoloured] film-forming component[s] (2a) that has a Dv.90 of no more than 75µm.

41. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 40], wherein there is [the uncoloured film-forming component or] at least one [of the] uncoloured [uncoloured] film-forming component[s] (2a) that has a Dv.99 in the range of from 50 to 65µm.

42. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 41], wherein there is [the uncoloured film-forming component or] at least one [of the] uncoloured [uncoloured] film-forming component[s] (2a) that has a Dv.50 in the range of from 5 to 45µm.

43. (Amended) The [A] powder coating composition of [as claimed in] claim 42, wherein there is [the uncoloured film-forming component or] at least one [of the] uncoloured [uncoloured] film-forming component[s] (2a) that has a Dv.50 of at least 8µm.

44. (Amended) The [A] powder coating composition of [as claimed in] claim 43, wherein there is [the uncoloured film-forming component or] at least one [of the] uncoloured [uncoloured] film-forming component[s] (2a) that has a Dv.99 of at least 40µm and a Dv.50 of at least 10µm

45. (Amended) The [A] powder coating composition of [as claimed in] claim 43 [or claim 44], wherein there is [the uncoloured film-forming component or] at least one [of the]

uncolored [uncoloured] film-forming component[s] (2a) that has a Dv.50 in the range of from 12 to 30µm.

46. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 45], wherein [the coloured film-forming base components or] at least one of the colored [coloured] film-forming base components (1) has [have] a Dv.99 in the range of from 6 to 25µm.

47. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 46], wherein [the coloured film-forming base components or] at least one of the colored [coloured] film-forming base components (1) has [have] a Dv.50 of no more than 18µm.

48. (Amended) The [A] powder coating composition of [as claimed in] claim 47, wherein [the coloured film-forming base components or] at least one of the colored [coloured] film-forming base components (1) has [have] a Dv.50 of no more than 15µm.

49. (Amended) The [A] powder coating composition of [as claimed in] claim 48, wherein [the coloured film-forming base components or] at least one of the colored [coloured] film-forming base components (1) has [have] a Dv.50 in the range of from 2 to 12µm.

50. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 49], wherein the colored [coloured] film-forming base components contain [in total] from 5 to 70 weight % of a pigment, calculated on the total weight of the colored film-forming base [those] components.

51. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 50], wherein the colored [coloured] film-forming base components contain [in total] from 0.5 to 50 weight % of the pigment, calculated on the total weight of the colored film-forming base [those] components.

52. (Amended) The [A] powder coating composition [as claimed in any one] of claim[s] 1 [to 51], wherein the individual particulate components of the agglomerate include one or more non-film-forming components.

53. (Amended) The [A] powder coating composition of [as claimed in] claim 52, wherein the individual particulate components of the agglomerate include a texturing agent.

54. (Amended) The [A] powder coating composition of [as claimed in] claim 52 [or claim 53], wherein the one or more non-film-forming [component or] components constitute up to 50% by weight of the [total] particulate components of the composition.

55. (Amended) A kit [comprising the following separate particulate components for agglomerating into powder coating compositions] for the preparation of powder coatings in a number of different colors comprising [colours]:

[●] a plurality of differently colored [coloured] film-forming base components, each with a Dv.99 of no more than 30µm, and

[●] one or more uncolored [uncoloured] film-forming components, each having a higher Dv.99[, and/or a higher Dv.50[.] than the colored film-forming base components and each Dv.99 being more than 40µm.

56. (Amended) The [A] kit of [as claimed in] claim 55, wherein there is [the film-forming component or] at least one [of the] uncolored [uncoloured] film-forming component[s] that has a Dv.99 of no more than 90µm.

57. (Amended) The [A] kit of [as claimed in] claim 55 [or claim 56], comprising [which includes] at least 7 differently colored [coloured] film-forming base components.

58. (Amended) The [A] kit [as claimed in any one] of claim[s] 55 [to 57], comprising [which includes] an uncolored [uncoloured] film-forming component that is compatible during film-formation with the colored [coloured] film-forming base components, and an

uncolored [uncoloured] film-forming component that is incompatible with the colored [coloured] film-forming base components or that becomes incompatible with the colored film-forming base components [therewith] during film-formation.

59. (Amended) A kit comprising [the following separate particulate components for the manufacture of agglomerated powder coating compositions] for the preparation of powder coatings in a number of different colors comprising [colours]:

- [•] at least 7 differently colored [coloured] film-forming base components,
- [•] an uncolored [uncoloured] film-forming component that is compatible with the colored [coloured] film-forming base components and remains compatible with the colored film-forming base components [therewith] during film-formation and that has a Dv.99 of more than 40µm and no more than 90µm, and
- [•] an uncolored [uncoloured] film-forming component that is incompatible with the colored [coloured] film-forming base components or that becomes incompatible with the colored film-forming base components [therewith] during film-formation[,] and that has a Dv.99 of more than 40µm and no more than 90µm.

60. (Amended) The [A] kit of [as claimed in] claim 59, comprising [which includes] means for comminution of the colored [coloured] film-forming base components to a powder having Dv.99 of no more than 30µm.

61. (Amended) The [A] kit [as claimed in any one] of claim[s] 55 [to 60], wherein the [different components are as specified in any one of claims 31 to 53] colored film-forming base components contain a polyester.

62. (Amended) The [A] kit [as claimed in any one] of claim[s] 55 [to 61], comprising [which includes] at least 3 uncolored [uncoloured] film-forming components.

63. (Amended) The [A] kit [as claimed in any one] of claim[s] 55 [to 62], comprising [which includes] means for agglomerating the components to produce a fluidisable powder.

64. (Amended) A process for preparing the [preparation of a] powder coating composition [as claimed in any one] of claim[s] 1[to 54], which comprises providing the [specified] one or more colored [coloured] film-forming base components (1) and the one or more [specified] uncolored [uncoloured] film-forming components (2a) [and if desired one or more other components selected from other uncoloured film-forming components (2b) and non-film-forming components (3), in the specified proportions,] to provide a mixture of components and mixing and agglomerating the mixture of components into composite particles such that the composition is air-fluidisable and can be applied to a substrate by electrostatic spray.

65. (Amended) A process for preparing the [preparation of a] powder coating composition of [as claimed in] claim 1 from a kit comprising a plurality of differently colored [coloured] film-forming base components and one or more uncolored [uncoloured] film-forming components having a Dv.99 of more than 40µm, which comprises comminuting at least one of the colored [coloured] film-forming base components to provide a powder having a Dv.99 of no more than 30µm, and mixing and agglomerating the at least one comminuted colored [coloured] film-forming base component [component(s)] and at least one [of the specified] uncolored [uncoloured] film-forming component having a Dv.99 or a Dv.50 higher than the Dv.99 or Dv.50 [respectively] of the at least one comminuted colored [coloured] film-forming base [component or] components taken together[, in the proportions specified in claim 1], respectively, to form composite particles, such that the composite particles are [composition is] air-fluidisable and can be applied to a substrate by electrostatic spray.

66. (Amended) The [A] powder coating composition [when] prepared by the [a] process of [as claimed in] claim 64 [or claim 65].

67. (Amended) A process for forming a coating on a substrate, which comprises applying the powder coating composition [an agglomerated composition as claimed in any one] of claim[s] 1 [to 54 or claim 66] to a substrate, and heating the applied composition to form a continuous coating.

68. (Amended) A substrate coated by a process of [as claimed in] claim 67.

POWDER COATING COMPOSITIONSFIELD OF THE INVENTION

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This invention relates to powder coating compositions and to their use.

Powder coating compositions generally comprise a solid film-forming resin, usually with one or more colouring agents such as pigments, and optionally they also contain one or more performance additives. They are usually thermosetting, incorporating, for example, a film-forming polymer and a corresponding curing agent (which may itself be another film-forming polymer).

The compositions are generally prepared by intimately mixing the ingredients (including any colouring agents and performance additives) for example in an extruder, at a temperature above the softening point of the film-forming polymer(s) but below a temperature at which significant pre-reaction would occur. The extrudate is usually rolled into a flat sheet and comminuted, for example by grinding to the desired particle sizes ("micronising"). The size distribution required for most commercial electrostatic spray apparatus is up to 120 microns, often between 10 and 120 microns, with a Dv.50 within the range of 15 to 75 microns, preferably 25 to 50 microns, or more especially 20 to 45 microns.

BACKGROUND TO THE INVENTION

Currently, powder coating manufacturing methods allow the manufacturer to offer commercially a range of full gloss coatings in a variety of colours. The range of products available in reduced-gloss finishes is, however, limited.

Reduction of gloss to some other lower level, for example satin gloss (55-65% gloss) or matt (< 30% gloss), is achieved by creating a surface which is rough on a microscopic scale. This may be achieved by using incompatible components or components that generate incompatibility. For example, an acrylic component and a polyester, epoxy, polyester-epoxy or polyurethane component are incompatible, and cannot be blended to form a single (stable) phase. Incompatibility during film formation can also be achieved by using components that are initially miscible (compatible) but that become incompatible during curing. Thus, for example, two systems of similar chemistry and approximately the same gel time are compatible, but components with different gel times are initially compatible but become incompatible as curing (and molecular weight build-up) proceeds. Thus, gloss-reducing agents include a second catalyst which will give a much faster gel time than the principal catalyst used to cure the film. Alternatively, with acid-functional polyesters, for example, a fast gelling (reacting) powder and a slow gelling powder may be manufactured separately using polyesters of different functionality, and mixed after the micronising stage or, more usually, the components are mixed prior to micronising; the components should have the same colour and particle size. Production of small batches of reduced-gloss coating composition, however, is uneconomic. A different method utilises a product called 'gloss killer', available from the company Tiger, which is mixed in after the micronising stage. However, this product, a clear (uncoloured) powder coating, can be added only in limited amounts to a conventional coloured powder coating before the presence of the gloss killer can be detected from the sparkle generated by the particles of the clear gloss killer powder in the film. Therefore the product is limited to adjusting gloss by a few percentage points.

There is accordingly a need for powder coating compositions in a wide range of reduced-gloss finishes, which avoid the problems mentioned above.

Speed of delivery to customers is also an important consideration, but the conventional powder coatings manufacturing method relies on premixing, extrusion and milling as separate processes, causing turnaround times to be long, and production of small batches of a product is again not economic. Stocking large product ranges solves the problem of speed of delivery, but is a highly inflexible approach and is not cost-effective.

EP 372860 A describes a colour mixing process for powder coatings in which sufficiently small-sized particles ($< 20 \mu\text{m}$ in size and advantageously $< 10 \mu\text{m}$) are used that mixed colours applied to a substrate have a homogeneous appearance.

10 Before application to the substrate the mixture is generally subjected to a process of agglomeration in which the small-sized particles are fused or bonded into composite particles, for example by mechanofusion, to convert the mixture from a cohesive mass to a free-flowing and fluidisable powder, which can be applied by conventional means. Thus, a range of basic coloured powder coating compositions is produced,

15 conventionally, in a conventional melt extrusion step, and the products are comminuted to a distinctively small particle size. A range of other colours can then be produced by mixing and agglomerating these coloured powder bases in the desired proportions. This allows the storage of comparatively few basic coloured powder bases, which can be mixed and agglomerated to produce any desired shade easily on demand, and the

20 production of small quantities becomes commercially feasible.

An extension of this agglomeration technique for the flexible production of a range of coloured powder coatings with a range of different reduced-gloss and other aesthetic effects is described in EP 539385 A.

Gloss reduction is achieved, for example, by adding uncoloured incompatible

25 particles of mean particle size $< 5\mu\text{m}$, or initially compatible particles of mean particle size $< 20\mu\text{m}$ of a polymer having a different functionality from the polymer of the main

film-forming component. Amounts of the gloss-reducing agent are, for example, 5 or 10% by weight, although amounts up to 40% by weight are also disclosed.

SUMMARY OF THE INVENTION

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The present invention provides a powder coating composition in which powder particles are an agglomerate of individual particulate components fused or bonded together into composite particles, wherein the individual particulate components comprise

- 10 (1) one or more coloured film-forming base components having a Dv.99 of no more than 30 μ m, and
- (2) one or more uncoloured film-forming components, wherein at least one component (2a)
- (i) has a Dv.99 that is more than 20 μ m and
- 15 (ii) has a higher Dv.99 or higher Dv.50 than component (1) or, when there is more than one such component, than those components taken together, the ratio of coloured film-forming base component(s) to uncoloured film-forming component(s) in the composition being in the range of from 1:99 to 60:40 by weight.

- As will be understood in the art, the volume percentiles Dv.x indicate for a
- 20 stated particle size (D) the percentage (x) of the total volume of the particles that lies below the stated particle size; the percentage (100-x) of the total volume lies at or above the stated size. Thus, for instance, Dv.50 is the median particle size of the sample, and on a particle size distribution graph Dv.99 is the point on the curve read along the particle size axis where the area under the curve below this particle size
- 25 represents 99% by volume of the particles. Thus, Dv.99 = 30 μ m indicates that 99% of the particles are below 30 μ m (but are not below 29 μ m). (For the avoidance of doubt, it

should be noted that *all* particle sizes quoted herein are by volume.) Volume percentiles are measurable by laser diffraction techniques, for example by the Malvern Mastersizer.

Surprisingly, even with a high proportion of uncoloured component of
5 significantly larger particle size than the coloured base components (whether judged according to Dv.99 or according to Dv.50), nevertheless the particles of the larger-sized component are not visible to the naked eye, so that the composition gives the appearance of a single colour.

A coloured base component comprises colouring material and film-forming
10 polymer. Thus, for example, it may be constituted as a powder coating composition in its own right, containing the usual powder coating additives, but having the specified (reduced) particle size. Usually there will be at least two coloured base components in the composition, each having a Dv.99 of no more than 30µm.

An uncoloured film-forming component is usually also a powder coating
15 composition in its own right, but having the specified particle size; there should be at least one uncoloured film-forming component having a higher Dv.99 and/or a higher Dv.50 than a coloured film-forming base component. (Usually, a component having a higher Dv.99 than another will also have a higher Dv.50 and vice-versa) Usually, there are two or more coloured film-forming base components each having a Dv.99 of no
20 more than 30µm and at least one uncoloured film-forming component having a Dv.99 that is more than 20µm and that is higher than the Dv.99 of the coloured film-forming base components taken together (or having a Dv.99 >20µm and a Dv.50 that is higher than the Dv.50 of the coloured film-forming base components taken together). Very often that uncoloured film-forming component will have a higher Dv.99 (or a higher
25 Dv.50) than each coloured film-forming base component. Preferably the uncoloured film-forming component has a Dv.99 of at least 40µm, for example at least 50µm, or

more than 55 μ m, e.g. in the range of from 50 to 65 μ m. Preferably it will have a Dv.90 of no more than 75 μ m and advantageously will have a Dv.50 of at least 8 μ m, especially in the range of from 12 to 30 μ m. Such uncoloured film-forming components have the advantage of lower manufacturing cost in relation to the small-sized coloured components and their use allows easy adjustment of the polymer/ pigment ratio in the composition.

The present invention also provides a kit comprising the following separate components for agglomerating into powder coating compositions for the preparation of powder coatings in a number of different finishes:

- 10 • a plurality of coloured film-forming base components, each with a Dv.99 of no more than 30 μ m, and
- one or more uncoloured film-forming components, at least one having a higher Dv.99 or Dv.50 than each of the coloured film-forming components, and a Dv.99 that is more than 20 μ m, preferably more than 40 μ m and preferably no more than 90 μ m.

Such kits allow a rapid and flexible manufacture of a range of coloured powder coating compositions, reduced-gloss finishes being manufactured by the use of an uncoloured component that is incompatible with the coloured base component or that becomes incompatible therewith during film-formation.

20 The present invention also provides a powder coating composition comprising a non-agglomerated mixture of the individual particulate components specified above in the specified proportions.

DETAILED DESCRIPTION OF THE INVENTION AND DESCRIPTION OF
PREFERRED EMBODIMENTS

The individual particulate components may comprise, for example,

- 5 (1) one or more, usually two or more, coloured film-forming base components each having a Dv.99 of no more than 30µm, in an amount of from 1 to 60% by weight of the total film-forming components, and
- (2)(a) one or more uncoloured film-forming components each having a Dv.99 of more than 20µm and each having a higher Dv.99 (or a higher Dv.50) than the
10 coloured film-forming base component (1) or, when there is more than one such component, than those components taken together, in an amount of from 10 to 99% by weight of the total film-forming components,
and, if desired,
- (b) one or more other uncoloured film-forming components, in an amount of up to
15 30% by weight of the total film-forming components,

Where there is one uncoloured film-forming component (2a) this preferably has a Dv.99 of up to 90µm, and where there are two or more uncoloured film-forming components (2a) preferably at least one, and often all, such components have a Dv.99 of up to 90µm.

- 20 More especially, the present invention provides a powder coating composition, suitable for providing a coating having certain appearance or performance attributes, which comprises composite particles formed by the agglomeration of individual particulate components fused or bonded together into composite particles such that the composite particles are air-fluidisable and can be applied to a substrate by electrostatic
25 spray without causing the individual particles in the composite particles to break down

under the mechanical and/or electrostatic forces associated with their application to a substrate, wherein the individual particulate components comprise

- (1) one or more, usually two or more, coloured film-forming base components each having a Dv.99 of no more than $30\mu\text{m}$ and
 - 5 (2)(a) one or more uncoloured film-forming components each having a Dv.99 of more than $20\mu\text{m}$ and each having a higher Dv.99 or Dv.50 than the coloured film-forming base component (1) or, when there is more than one such component, than those components taken together,
- and, if desired,
- 10 (b) one or more other uncoloured film-forming components,
- the ratio of coloured film-forming base component(s) to uncoloured film-forming component(s) in the composition being in the range of from 1:99 to 60:40 by weight, each particle of each film-forming component comprising a solid polymeric binder system at least a portion of which is a film-forming resin, the resin in the composition
- 15 being in an amount sufficient to impart film-forming properties to the composition.

Ratios of coloured film-forming component(s) to uncoloured film-forming component(s) in the composition of from 1:99, e.g. from 2:98, ~~preferably~~ preferably from 5:95, and up to 60:40, e.g. no more than 50:50, advantageously no more than 40:60 and preferably no more than 30:70, should especially be mentioned.

- 20 Advantageously, the minimum weight of uncoloured film-forming component(s) (2a) corresponds to a ratio of coloured film-forming base component(s) (1) to uncoloured film-forming component(s) (2a) of 5:2. Preferably the weight of uncoloured film-forming component(s) (2a) is at least that of coloured film-forming component(s) (1) and, preferably, the weight of uncoloured film-forming component(s) (2a) is at least
- 25 that of other uncoloured film-forming component(s). Preferably the weight of

uncoloured film-forming component(s) (2a) is at least that of other film-forming component(s) (1) and (2b) together.

In one embodiment, the uncoloured film-forming component(s) (2a) have a Dv.99 of more than $20\mu\text{m}$ and up to $40\mu\text{m}$, the ratio of coloured film-forming base component(s) to uncoloured film-forming component(s) in the composition being in the range of from 1:99 to 30:70, for example from 2:98 to 30:70, by weight.

In another embodiment, the uncoloured film-forming component(s) have a Dv.99 of more than $40\mu\text{m}$ and preferably no more than $90\mu\text{m}$, the ratio of coloured film-forming base component(s) to uncoloured film-forming component(s) in the composition being in the range of from 1:99 to 60:40, for example from 2:98 to 30:70, by weight.

Thus, the coloured film-forming base component(s) (1) may be, for example, at least 2%, for example at least 5%, and, for example, no more than 50%, advantageously no more than 40% and preferably no more than 30%, by weight of the total film-forming components. A content of coloured film-forming base component(s) of no more than 20%, for example from 10 to 20%, by weight of the total film-forming components should especially be mentioned.

The uncoloured film-forming component(s) (2) may be, for example, at least 10%, preferably at least 20%, more preferably at least 30%, especially at least 40%, more especially at least 50%, advantageously at least 60%, for example at least 70%, and, for example, up to 95%, by weight of the total film-forming components. A content of uncoloured film-forming component(s) of up to 90%, for example from 80 to 90%, by weight of the total film-forming components should be mentioned.

The uncoloured film-forming component(s) (2a) may be, for example, at least 10%, preferably at least 20%, more preferably at least 30%, especially at least 40%,

more especially at least 50%, advantageously at least 60%, for example at least 70%, and for example up to 95%, by weight of the total film-forming components.

Where the composition also includes, as particulate component in the agglomerate, a further uncoloured film-forming component (component (2b)), there may, for example, be no more than 30% by weight, calculated on all of the film-forming components together, of component(s) (2b) having (i) a Dv.99 that is $\leq 20\mu\text{m}$, and (ii) a Dv.99 (or Dv.50) that is no higher than the Dv.99 (or Dv.50 respectively) of the coloured film-forming component or of all the coloured film-forming components in the composition taken together. A content of uncoloured film-forming component(s) (2b) of no more than 15%, for example 10% by weight or less, calculated on the weight of the total film-forming components should especially be mentioned.

In comparison with agglomerated coloured powder coating compositions described in EP 372860 A and EP 539385 A, compositions of the present invention contain a high proportion of uncoloured film-forming component, and the individual coloured components necessarily contain a significantly higher content of pigment. Moreover, the particle size of the uncoloured particles in component(s) (2a) can be significantly larger than those of the coloured base component(s); without, surprisingly, affecting visual homogeneity. Thus, contrary to expectation and contrary to the suggestions in EP 372860 A and EP 539385 A, powders containing significant quantities of uncoloured component(s) having, for example, a Dv.99 $> 40\mu\text{m}$ can be used to produce powder coating films in which the differences between the coloured particles and the uncoloured particles are not discernable by the unaided human eye. Maximising the level of larger-sized uncoloured component(s) will give cost advantages.

If desired, the composition may include as particulate component in the agglomerate as well as coloured film-forming base component(s) (1) and uncoloured

film-forming component(s) (2a) and optionally (2b), one or more non-film-forming components, e.g. a texturing agent, a metallic or mica pigment, or a non-film-forming performance component. If desired, a non-film-forming component may be pre-mixed with a film-forming component, e.g. with an uncoloured film-forming component, to form a masterbatch before mixing with the remaining components and then agglomerating. Alternatively, for example, a non-film-forming additive may be mixed with an uncoloured film-forming composition in an extruder and then micronised, to give an uncoloured film-forming component (2a) or (2b), depending on particle size, that also contains the desired aesthetic or performance additive.

- 10 A film-forming particulate component for inclusion in the agglomerate may itself be in the form of an agglomerate. Usually such a component would be formed by agglomeration of an uncoloured film-forming component with a non-film-forming component, for example a texturing additive or metallic or mica pigment.

- Each film-forming component of the composition comprises at least one solid film-forming resin and includes any curing agent required therefor, and is usually formed by an extrusion process and comminution to the requisite particle size. Where a film-forming component is coloured, the colouring agent or agents (pigments and/or dyes) is (are) generally extruded with the film-forming resin(s), plus any curing agent, so that particles formed therefrom comprise film-forming resin, colouring agent and, where applicable, curing agent.

- Reduction of gloss, for example to satin gloss (55-65% gloss) or matt (<30% gloss) or some other level, may be achieved by creating a surface which is rough on a microscopic scale using incompatible components or components that generate incompatibility. Film-forming components of similar chemistry but different gel times provide incompatibility during film-formation, and reduction in gloss may be achieved by such means. Usually two (or more) film-forming base components are compatible with each other, but there may be an incompatible uncoloured film-forming component

present. For example, there may be an uncoloured film-forming component having a different functionality from the coloured film-forming base component(s) and optionally also containing a different catalyst (both of which lead to different gel times). For example, coloured and uncoloured components may be based on polyesters of different functionality. For polyurethane systems using hydroxy-functional polyesters cured with an isocyanate (typically isophorone diisocyanate), hydroxy-functional polyesters with radically different functionality may be used, e.g. a hydroxy-functional polyester with a functionality of 7 for an uncoloured component and one with a functionality of 2 used for the coloured base component(s). Another possibility is to employ polymeric materials that are *per se* incompatible with each other, for example a polyester and an acrylic polymer.

In contrast to conventional prior art gloss-reduction processes, which generally use 50:50 non-fused mixtures of same-sized powders, with fused or bonded compositions of the present invention good gloss reduction can be achieved with substantially different proportions of gloss-reducing additive. The agglomerated material also does not suffer from particle segregation in the solid state as would dry-blended product, thus giving uniformity of product even after transportation and spraying.

Thus, uncoloured components can be used for extension of product ranges, to give different finishes. Also, by using different chemistries, they may be used to give different performance characteristics. The different chemistries may arise, for example, from the use of different film-forming polymers, e.g. polyester and acrylic or polyester and epoxy, but may also arise from the use of different curing agents, e.g. polyester with an epoxy curing agent in one component and polyester with a bis(beta-hydroxyalkylamide) curing agent such as PRIMID in another component.

In addition, or alternatively, an uncoloured film-forming component of the composition of the present invention may be compatible with the coloured film-forming

base component(s). Usually all the coloured film-forming base components are compatible with one another.

- An uncoloured film-forming component of a kit of the invention may be a pre-prepared uncoloured coating composition of the specified particle size or one of conventional size which is reduced in size just prior to use. Advantageously, for any particular type of film-forming chemistry (e.g. acid-functional polyester, hydroxy-functional polyester), a kit of the invention includes a "universal" gloss-reducing component suitable for all powder coating compositions of that chemistry. The present invention has the advantage of reducing costs while also reducing stocking levels and manufacturing capacity. It enables a very rapid and flexible service to be provided cheaply to the customer, allowing for the possibility of providing small quantities of powder coating compositions economically on request.

- Advantageously, the coloured film-forming base components taken together contain at least 5%, e.g. at least 10%, and generally no more than 70% or 75%, e.g. no more than 60%, for example 20 to 40%, by weight of pigment, calculated on the total weight of these components. Advantageously, each contains at least 8% by weight of pigment, calculated on the weight of that component. The number of coloured film-forming base components in a kit may be, for example, at least 7, e.g. in the range of from 7 to 30.

- Examples of pigments which may be used in the coloured base components are inorganic pigments, such as, for example, titanium dioxide white, red and yellow iron oxides, chrome pigments and carbon black, and organic pigments such as, for example, phthalocyanine, azo, anthraquinone, thioindigo, isodibenzanthrone, triphenyldioxane and quinacridone pigments, vat dye pigments and lakes of acid, basic and mordant dyestuffs. Dyes may be used instead of or as well as pigments. Each coloured base component of the coating composition may contain a single colorant

(pigment or dye) or may contain more than one colorant. Where appropriate, a filler may be used to assist opacity, whilst minimising costs.

Additional pigment may alternatively be added as a separate component prior to agglomerating, especially if the pigment is close in colour to the mixed coloured film-forming base components. Any pigment added in this way would generally be no more than 3%, preferably up to 1%, by weight, based on the weight of the total composition, although amounts up to 5% may also be possible. Thus, for example, pigment in an amount of no more than 3% by weight, more especially up to 1% by weight, may be used to displace the colour of the components over a small colour region, this being used for colour tinting or colour correction of a batch.

Preferably, the total weight of pigment in the composition is at least 0.5%, more especially at least 5%, and preferably no more than 30%, although an amount up to 50% is potentially also possible.

One or more other separate components (3) may also be present. Other optional components include, for example, performance and aesthetic additives mentioned in EP 539385 A. Those components may, if desired, be pre-mixed with another component, for example an uncoloured film-forming component, with the components remaining as distinct components, e.g. (2a) and (3), before final agglomeration. Alternatively, the additives may be combined in a masterbatch with uncoloured film-forming polymer, often itself constituted as a powder coating composition in its own right, the masterbatch being prepared, for example, by co-extrusion of polymer and additive, followed by comminution, or by agglomeration of the particulate additive with uncoloured film-forming powder, to form uncoloured film-forming component (2a) or (2b), often (2a). Such further aesthetics additives may be present for example in an amount of from 0.5 to 50% by weight of the total composition. A non-film-forming performance additive is generally present in an amount of no more than 5% by weight of the composition, e.g. in an amount of from 0.5

to 5% by weight. Where other component(s) are present in a composition of the present invention, the percentages of the specified coloured film-forming base component(s) and/or the specified uncoloured film-forming component(s) in the composition may be altered accordingly, but the ratio of coloured film-forming base component(s) to uncoloured film-forming component(s) will generally remain the same.

A composition of the present invention may contain, for example, at least 1%, e.g. at least 2%, often at least 5%, by weight of the specified coloured film-forming base component(s). Often the composition will contain at least 20%, e.g. at least 30% or at least 40%, by weight of the uncoloured film-forming component (2a) of the size specified above. Preferably, the content of uncoloured film-forming component(s) in the composition is at least 40%, e.g. at least 50%, and advantageously at least 60%, and preferably at least 70%, by weight. Amounts of at least 40% e.g. at least 50%, and advantageously at least 60%, and preferably at least 70%, by weight of uncoloured film-forming component(s) (2a) of the size specified above should especially be mentioned.

Ratios of from 1:99, e.g. from 2:98, preferably from 5:95, and up to 60:40, e.g. up to 50:50, advantageously up to 40:60 and preferably up to 30:70, for the ratio of coloured film-forming base component(s) (1) to uncoloured film-forming component(s) of the specified size (2a) should especially be mentioned.

Preferably, in each of the coloured film-forming base components all the component particles are $< 25\mu\text{m}$. Advantageously the coloured film-forming base components have at least 90% by volume of particles < 20 microns, more especially at least 90% by volume < 10 microns, and advantageously the components have a Dv.99 of at least $6\mu\text{m}$, advantageously up to $25\mu\text{m}$. Advantageously, the Dv.50 of each such component is up to $18\mu\text{m}$, preferably up to $15\mu\text{m}$, e.g. up to $12\mu\text{m}$, advantageously at

least $2\mu\text{m}$, for example within the range of for example 2 to $8\mu\text{m}$, preferably 2 to $6\mu\text{m}$ or 8 to $12\mu\text{m}$; Dv.50 values $\geq 2\mu\text{m}$ and $\leq 5\mu\text{m}$ should especially be mentioned.

A coloured film-forming base component of a kit of the invention may, however, be of larger particle size and then reduced in size just prior to use. Accordingly, the present invention also provides a kit comprising the following separate components for agglomerating into powder coating compositions for the preparation of powder coatings in a number of different colours:

- at least 7 differently coloured film-forming base components,
- an uncoloured film-forming component that is compatible with the coloured film-forming base components and remains compatible therewith during film-formation and that has a Dv.99 of more than $40\mu\text{m}$ and preferably of no more than $90\mu\text{m}$, and
- an uncoloured film-forming component that is incompatible with the coloured film-forming base components or that becomes incompatible therewith during film-formation and that has a Dv.99 of more than $40\mu\text{m}$ and preferably of no more than $90\mu\text{m}$.

In one embodiment of the present invention, the individual particulate components of the powder coating composition comprise

- two or more coloured film-forming base components having a Dv.99 of no more than $30\mu\text{m}$, and
- one or more uncoloured film-forming components, at least one component having a higher Dv.99 or higher Dv.50 than at least one, preferably all, of the coloured film-forming base component(s), and having a Dv.99 of more than $20\mu\text{m}$ and preferably no more than $90\mu\text{m}$,

the ratio of coloured film-forming base components to uncoloured film-forming component(s) of the specified size being in the range of from 1:99 to 30:70 by weight.

In a further embodiment of the present invention, the individual particulate components of the powder coating composition comprise

- two or more coloured film-forming base components having a Dv.99 of no more than $30\mu\text{m}$, and
- one or more uncoloured film-forming components, at least one component having a Dv.99 of more than $40\mu\text{m}$ and preferably no more than $90\mu\text{m}$, the ratio of coloured film-forming base components to uncoloured film-forming component(s) of the specified size being in the range of from 1:99 to 60:40 by weight.

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Preferably, the uncoloured film-forming component (2a) or at least one, and advantageously each, of the uncoloured film-forming components (2a) has a Dv.99 of at least $30\mu\text{m}$, more especially at least $35\mu\text{m}$, and advantageously at least $40\mu\text{m}$. An uncoloured film-forming component having a Dv.50 of at least $8\mu\text{m}$ and up to $35\mu\text{m}$, e.g. up to $30\mu\text{m}$, and/or having a Dv.90 of at least $14\mu\text{m}$ should especially be mentioned. An uncoloured film-forming component (2a) having a Dv.50 of at least $10\mu\text{m}$ and up to $35\mu\text{m}$, e.g. in the range of from 12 to $30\mu\text{m}$, and/or having a Dv.90 of at least $18\mu\text{m}$ and one having a Dv.90 of no more than $75\mu\text{m}$ should also especially be mentioned.

Uncoloured component(s) (2a) may, for example, have a Dv.99 or a Dv.50 that is at least twice, e.g. at least three times, the Dv.99 or Dv.50 figure of the coloured component(s) (1) taken together. For example, the Dv.99 of component(s) (2a) may be at least $20\mu\text{m}$, e.g. at least $30\mu\text{m}$, more than that of component or components (1) taken together.

A powder coating composition of the invention will in general be a thermosetting system, although thermoplastic systems (based, for example, on polyamides) can in principle be used instead.

When a thermosetting resin is used, the solid polymeric binder system generally includes a solid curing agent for the thermosetting resin; alternatively two co-reactive film-forming thermosetting resins may be used. Thus, a thermosetting powder coating composition according to the invention may contain one or more film-forming polymers selected from carboxy-functional polyester resins, hydroxy-functional polyester resins, epoxy resins, and functional acrylic resins. Carboxy-functional polyester resins should especially be mentioned.

A carboxy-functional polyester film-forming resin may be used, for example, with a polyepoxide curing agent. Such carboxy-functional polyester systems are currently the most widely used powder coatings materials. The polyester generally has an acid value in the range 10-100, a number average molecular weight M_n of 1,500 to 10,000 and a glass transition temperature T_g of from 30°C to 85°C, preferably at least 40°C. The poly-epoxide can, for example, be a low molecular weight epoxy compound such as triglycidyl isocyanurate (TGIC), a compound such as diglycidyl terephthalate condensed glycidyl ether of bisphenol A or a light-stable epoxy resin. Such a carboxy-functional polyester film-forming resin can alternatively be used with a bis(beta-hydroxyalkylamide) curing agent such as tetrakis(2-hydroxyethyl)adipamide.

Alternatively, a hydroxy-functional polyester can be used with a blocked isocyanate-functional curing agent or an amine-formaldehyde condensate such as, for example, a melamine resin, a urea-formaldehyde resin, or a glycol ural formaldehyde resin, for example the material "Powderlink 1174" supplied by the Cyanamid Company, or hexahydroxymethyl melamine. A blocked isocyanate curing agent for a hydroxy-functional polyester may, for example, be internally blocked, such as the uret dione type, or may be of the caprolactam-blocked type, for example isopherone diisocyanate.

As a further possibility, an epoxy resin can be used with an amine-functional curing agent such as, for example, dicyandiamide. Instead of an amine-functional

curing agent for an epoxy resin, a phenolic material may be used, preferably a material formed by reaction of epichlorohydrin with an excess of bisphenol A (that is to say, a polyphenol made by adducting bisphenol A and an epoxy resin). A functional acrylic resin, for example a carboxy-, hydroxy- or epoxy-functional resin can be used with an appropriate curing agent.

Mixtures of film-forming polymers can be used; for example a carboxy-functional polyester can be used with a carboxy-functional acrylic resin and a curing agent such as a bis(beta-hydroxyalkylamide) which serves to cure both polymers. As further possibilities, for mixed binder systems, a carboxy-, hydroxy- or epoxy-functional acrylic resin may be used with an epoxy resin or a polyester resin (carboxy- or hydroxy-functional). Such resin combinations may be selected so as to be co-curing, for example a carboxy-functional acrylic resin co-cured with an epoxy resin, or a carboxy-functional polyester co-cured with a glycidyl-functional acrylic resin. More usually, however, such mixed binder systems are formulated so as to be cured with a single curing agent (for example, use of a blocked isocyanate to cure a hydroxy-functional acrylic resin and a hydroxy-functional polyester). Another preferred formulation involves the use of a different curing agent for each binder of a mixture of two polymeric binders (for example, an amine-cured epoxy resin used in conjunction with a blocked isocyanate-cured hydroxy-functional acrylic resin).

Other film-forming polymers which may be mentioned include functional fluoropolymers, functional fluorochloropolymers and functional fluoroacrylic polymers, each of which may be hydroxy-functional or carboxy-functional, and may be used as the sole film-forming polymer or in conjunction with one or more functional acrylic, polyester and/or epoxy resins, with appropriate curing agents for the functional polymers.

Other curing agents which may be mentioned include epoxy phenol novolacs and epoxy cresol novolacs; isocyanate curing agents blocked with oximes, such as

- isophorone diisocyanate blocked with methyl ethyl ketoxime, tetramethylene xylene diisocyanate blocked with acetone oxime, and Desmodur W (dicyclohexylmethane diisocyanate curing agent) blocked with methyl ethyl ketoxime; light-stable epoxy resins such as "Santolink LSE 120" supplied by Monsanto; and alicyclic poly-epoxides
- 5 such as "EHPE-3150" supplied by Daicel.

- The function of coatings is of course protective, but appearance is also important, and the film-forming resin and other ingredients are selected so as to provide the desired performance and appearance characteristics. In relation to performance, coatings should generally be durable and exhibit good weatherability,
- 10 stain or dirt resistance, chemical or solvent resistance and/or corrosion resistance, as well as good mechanical properties, e.g. hardness, flexibility or resistance to mechanical impact; the precise characteristics required will depend on the intended use. The composition must, of course, be capable of forming a coherent film on the substrate, and good flow and levelling of the composition on the substrate are required.
- 15 Accordingly, the powder coating composition generally also contains one or more performance additives such as, for example, a flow-promoting agent, a plasticiser, a stabiliser, for example a stabiliser against UV degradation, an anti-gassing agent, such as benzoin, or a filler. Such additives are known and standard additives for use in powder coating compositions. Usually, these performance additives will be
- 20 incorporated in any film-forming component before and/or during the extrusion or other homogenisation process, although, if appropriate, any such additive may alternatively be incorporated as a separate component in the agglomeration process.

Mixing and agglomeration methods are described in EP 372860 A and EP 539385 A.

- 25 The agglomerate may, for example, be prepared by mechanofusion of a mixture of the individual components, for example by mechanofusion at a temperature in the range of from 60 to 80°C, or by granulation using methanol or other suitable solvent as

granulating agent, to produce composite particles that constitute a free-flowing and fluidisable powder.

Good fluidity of the powder is required for purposes of application to the substrate. This fluidity of powders is governed by their particle size, and particle size
5 also controls the application efficiency of a powder; powders with small particles, i.e. significant quantities <10 microns, more especially <5 microns, exhibit poor fluidity and application characteristics.

The present invention also provides a process for the preparation of a powder coating composition of the present invention, which comprises providing, in the
10 specified proportions, the specified one or more coloured film-forming base components and the one or more specified uncoloured film-forming components, and if desired one or more other uncoloured film-forming components and/or one or more non-film-forming components, and mixing and agglomerating the components such that the composition is air-fluidisable and can be applied to a substrate by electrostatic
15 spray.

Agglomeration prevents segregation of the constituents during application and handling, which otherwise would occur, for example during the application process itself (because of differential electrostatic charging) or in the attendant recovery and recycling process (because of differences in particle size and/or particle density) or in
20 transport, causing batch-to-batch variability in the resulting coating.

The composite particles produced may be likened to raspberries with the individual particles of the raspberry (the drupels) bonded to one another, although the "drupels" are of different sizes, and there will of course also be "raspberries" of different composition and different sizes in the powder as a whole. Inspection of a fused
25 agglomerated powder under an electron microscope shows that one particle is bonded to another and that the individual particles in the composites are more rounded than prior to agglomeration. When a conventional powder coating composition is inspected

under an electron microscope, however, the powder particles are seen to be sharp-edged or angular, and are seen as separate, distinct particles - they are not fused to one another.

- Furthermore, unlike conventional uniformly coloured powder coating compositions, that contain particles of only a single colour (produced by fusion in the melt extruder), powder coating compositions of this invention consist of a mixture of differently coloured particles and uncoloured particles but nevertheless give the appearance of a single colour on application to a substrate.

- An agglomerated powder coating composition according to the invention may in principle be applied to a substrate by any suitable process of powder coating technology, for example by electrostatic spray coating, or by fluidised-bed or electrostatic fluidised-bed processes.

- After application of the powder coating composition to a substrate, conversion of the resulting adherent particles into a continuous coating (including, where appropriate, curing of the applied composition) may be effected by heat treatment and/or by radiant energy, notably infra-red, ultra-violet or electron beam radiation.

- The powder is usually cured on the substrate by the application of heat (the process of stoving), usually for a period of from 5 to 30 minutes and usually at a temperature in the range of from 150 to 220°C, although temperatures down to 90°C may be used for some resins, especially epoxy resins; the powder particles melt and flow and a film is formed. The curing times and temperatures are interdependent in accordance with the composition formulation that is used, and the following typical ranges may be mentioned:

<u>Temperature/°C</u>	<u>Time</u>
280 to 100*	10 s to 40 min
250 to 150	15 s to 30 min
220 to 160	5 min to 20 min

- 5 * Temperature down to 90°C may be used for some resins, especially certain epoxy resins.

The invention also provides a process for forming a coating on a substrate, which comprises applying an agglomerated composition according to the invention to a substrate, for example by an electrostatic spray coating process, and heating the
10 applied composition to melt and fuse the particles and where appropriate cure the coating.

The film may be any suitable thickness. For decorative finishes, film thicknesses as low as 20 microns should be mentioned, but it is more usual for the film thickness to fall within the range 25-120 microns, with common ranges being 30-80
15 microns for some applications, and 60-120 microns or, more preferably, 60-100 microns for other applications, while film thicknesses of 80-150 microns are less common, but not rare.

The substrate may comprise a metal, a heat-stable plastics material, wood, glass, or a ceramic or textile material. Advantageously, a metal substrate is chemically
20 or mechanically cleaned prior to application of the composition, and is preferably subjected to chemical pre-treatment, for example with iron phosphate, zinc phosphate or chromate. Substrates other than metallic are in general preheated prior to application or, in the case of electrostatic spray application, are pretreated with a material that will aid such application.

The following Examples illustrate the invention. In each case the particle sizes were measured on the Malvern Mastersizer X laser light-scattering device from Malvern Instruments.

5 EXAMPLES

Preparation of Individual Components

- White, black and red coloured film-forming base components and different
10 uncoloured film-forming components were prepared by mixing the following formulations.

Component A - White Powder Coating Composition

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| 15 | Rutile titanium dioxide white pigment | 600 g |
| | Carboxylic acid-functional polyester resin | 360 g |
| | Bis(beta-hydroxyalkylamide) curing agent | 13 g |
| | Benzoin degassing agent | 4 g |
| | Flow modifiers | 18 g |
| 20 | Surface waxes | 5 g |

Component B - Black Powder Coating Composition

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|----|--|-------|
| | Carbon Black pigment | 175 g |
| 25 | Carboxylic acid-functional polyester resin | 772 g |
| | Bis(beta-hydroxyalkylamide) curing agent | 27 g |
| | Benzoin degassing agent | 4 g |

Flow modifiers	18 g
Surface waxes	4 g

Component C - Red Powder Coating Composition

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Earth Red iron oxide pigment	125 g
Carboxylic acid-functional polyester resin	650 g
Bis(beta-hydroxyalkylamide) curing agent	24 g
Benzoin degassing agent	2 g
10 Flow modifiers	18 g
Surface waxes	4 g
Fillers (barytes)	177 g

For the coloured components A to C the ingredients were dry mixed and fed to

15 an extruder blender operating at a temperature of 100°C. The extruder produced a sheet of pigmented resin which was ground to a particle size of below 100 µm and milled on a 100 AFG jet-mill (manufacturer Hosakawa Micron) at 6 bar grinding air pressure and classified at a speed of 7000 rpm.

The particle size distribution for Components A, B and C was:

20 Dv.50 = 3.5µm
 Dv.90 = 5.9µm
 Dv.99 = 8.2µm

The polyester of Components A to C had an acid value of 18 to 30.

Component D - Uncoloured Powder Coating Composition

	Carboxylic acid-functional polyester resin	650 g
	Bis(beta-hydroxyalkylamide) curing agent	24 g
5	Benzoin degassing agent	2 g
	Flow modifiers	18 g
	Surface waxes	4 g
	Fillers (barytes)	302 g

10 Component E - Uncoloured Powder Coating Composition for gloss-reduction

	Carboxylic acid-functional polyester resin	575 g
	Fillers (barytes)	333 g
	Benzoin degassing agent	4 g
15	Surface waxes	4 g
	Flow modifiers	18 g
	Bis(beta-hydroxyalkylamide) curing agent	65 g

- For uncoloured components D and E the procedure for components A to C was repeated except that the jet-milling was carried out at 3000 rpm.

The particle size distribution for components D and E is:

$$Dv.50 = 18.4\mu m$$

$$Dv.90 = 36.2\mu m$$

$$Dv.99 = 51.0\mu m$$

- 25 The polyester of Component D had an acid value of 18 to 30 and the polyester of Component E had an acid value of 40 to 90.

Component F - Uncoloured Powder Coating Composition for Texturing

	Carboxylic acid-functional polyester resin	780 g
5	Texturing agent: (PTFE wax, silicone wax, cellulose acetyl butyrate, ester modified polyether	220 g

The ingredients were dry mixed and fed to an extruder blender operating at a temperature of 100°C. This was ground to a powder in an air classified impact mill.

10 The particle size distribution for component F is

Dv.50 = 36µm

Dv.90 = 82µm

Dv.99 = 118µm

The polyester of Component F had an acid value of 18 to 30.

15

Component G - Uncoloured Powder Coating Composition for Pearlescent Effects

	Component E	800 g
	Mica Pigment (e.g. Iriodin 9111 supplied by Merck Ltd., Poole, Dorset, England)	200 g
20		

The ingredients were blended together in a Henschel FM10 mixer for 30 minutes in total, taking the temperature to 54°C. The agglomerated powder was sieved through a 110µm steel mesh.

25 The particle size distribution for component G is

Dv.50 = 13µm

$$Dv.90 = 24\mu m$$

$$Dv.99 = 38\mu m$$

Components H and I

5

Components A and B were prepared as before but milled on a 100 AFG jet-mill at 6 Bar grinding air pressure and classified at a speed of 12000rpm. These components were re-labelled Components H and I respectively.

The particle size distribution for Components H and I were:

10

$$Dv.50 = 2.6\mu m$$

$$Dv.90 = 5.3\mu m$$

$$Dv.99 = 6.4\mu m$$

Component J - Uncoloured Powder Coating composition for Tribo Application

15

Carboxylic acid-functional polyester resin	875 g
Bis-(beta-hydroxyalkylamide) curing agent	50 g
Benzoin degassing agent	2 g
Flow modifier	26 g
20 Tribostatic additives	47 g

The ingredients were dry mixed and fed to an extruder blender operating at a temperature of 100°C. The extruder produced a sheet of uncoloured resin which was ground to a particle size of below 100 μm and milled on a 100 AFG jet-mill (manufacturer Hosakawa Micron) at 6 Bar grinding air pressure and classified at a speed of 6500 rpm.

25

The particle size distribution for Component J is

$$Dv.50 = 5.3\mu m$$

$$Dv.90 = 8.2\mu m$$

5

$$Dv.99 = 9.4\mu m$$

The polyester of component J had an acid value of 18 to 30.

Component K - Uncoloured Powder Coating Composition (Polyester/Epoxy)

10

Carboxylic acid-functional polyester resin 440 g

Glycidyl-functional Bisphenol-A epoxy resin

(type II or type III epoxy) 290 g

Benzoin degassing agent 2 g

15 Flow modifiers 18 g

Fillers (barytes) 250 g

The ingredients were dry mixed and fed to an extruder blender operating at a temperature of 100°C. This was ground to a powder in an air classified impact mill.

20

The particle size distribution for component K is

$$Dv.50 = 28\mu m$$

$$Dv.90 = 58\mu m$$

$$Dv.99 = 72\mu m$$

The polyester of Component K had an acid value of 18 to 30.

25

Preparation and Use of Agglomerated Powder Coating Compositions

Example 1

5 A mixture comprising:

Component A	187.5 g
Component B	16.7 g
Component C	100.0 g
Component D	695.8 g

10 was blended together in a Henschel FM10 mixture for 30 minutes in total, with a water jacket taking the temperature to 54°C. The agglomerated powder was sieved through a 110µm steel mesh and then electrostatically applied through a Gema PCG-1 corona spray gun onto an aluminium Q panel. This was then stoved at the recommended time and temperature.

15 A smooth, glossy coating that had a homogeneous dusky pink colour was produced.

Example 2

20 A mixture comprising

Component A	187.5 g
Component B	16.7 g
Component C	100.0 g
Component D	545.8 g

25 Component E 150.0 g

was agglomerated and applied to a substrate and stoved as in Example 1.

As shown by their acid numbers, the polyester of Component E has a different functionality and hence different gel time from components A to D, and a smooth, matt coating of 30% gloss at 60° angle is produced that has a homogeneous dusky pink colour.

5

Example 3

A mixture comprising

10	Component A	65 g
	Component B	75 g
	Component C	80 g
	Component D	760 g
	Component F	20 g

was blended together in a Henschel mixer for 30 minutes in total, taking the
15 temperature to 54°C. The agglomerated powder was sieved through a 110µm steel mesh and then electrostatically applied through a Gema PCG-1 corona spray gun onto an aluminium Q Panel. This was then stoved at the recommended time and temperature.

20 A textured, glossy coating that had a homogeneous dark brown colour was produced.

Example 4

A mixture comprising

25	Component A	250 g
	Component B	125 g

Component D 475 g

Component G 150 g

was blended together in a Henschel mixer for 30 minutes in total, taking the temperature to 54°C. The agglomerated powder was sieved through a 110µm steel mesh and then electrostatically applied through a Gema PCG-1 corona spray gun onto an aluminium Q Panel. This was then stoved at the recommended time and temperature.

A smooth, matt coating that had a homogeneous grey colour and a pearlescent metallic effect was produced.

Example 5

A mixture comprising

Component H 40 g

Component I 120 g

Component D 670 g

Component E 70 g

Component J 100 g

was blended together in a Henschel mixer for 30 minutes in total, taking the temperature to 54°C. The agglomerated powder was sieved through a 110µm steel mesh and then electrostatically applied by a tribostatic gun onto an aluminium Q Panel. This was then stoved at the recommended time and temperature.

A smooth, satin coating that had a homogeneous charcoal colour was produced.

Example 6

A mixture comprising:

	Component A	187.5 g
5	Component B	16.7 g
	Component C	100.0 g
	Component K	695.8 g

was blended together in a Henschel FM10 mixture for 30 minutes in total, with a water jacket taking the temperature to 54°C. The agglomerated powder was sieved through a 110µm steel mesh and then electrostatically applied through a Gema PCG-1 corona spray gun onto an aluminium Q panel. This was then stoved at the recommended time and temperature.

A smooth, glossy coating that had a homogeneous dusky pink colour was produced.

CLAIMS

1. A powder coating composition in which powder particles are an agglomerate of individual particulate components fused or bonded together into composite particles, wherein the individual particulate components comprise
- 5 (1) one or more coloured film-forming base components having a Dv.99 of no more than $30\mu\text{m}$, and
- (2) one or more uncoloured film-forming components, wherein at least one component (2a) (i) has a Dv.99 that is more than $20\mu\text{m}$ and (ii) has a higher
- 10 Dv.99 or higher Dv.50 than component (1) or, when there is more than one coloured component, than those components taken together,
- the ratio of coloured film-forming base component(s) to uncoloured film-forming component(s) in the composition being in the range of from 1:99 to 60:40 by weight.
- 15 2. A powder coating composition as claimed in claim 1, wherein the uncoloured film-forming component or components (2a) are present in an amount of at least 10% by weight of the total film-forming components.
3. A powder coating composition as claimed in claim 1 or claim 2, wherein
- 20 there is no more than 30% by weight, calculated on the weight of the total film-forming components, of uncoloured film-forming component(s) having a Dv.99 (i) that is no higher than the Dv.99 of the coloured film-forming component or components taken together, and (ii) that is less than $20\mu\text{m}$.

4. A powder coating composition in which powder particles are an agglomerate of individual particulate components fused or bonded together into composite particles, wherein the individual particulate components comprise

(1) one or more coloured film-forming base components each having a Dv.99 of no more than 30µm, in an amount of from 1 to 60% by weight of the total film-forming components, and

(2)(a) one or more uncoloured film-forming components each having a Dv.99 of more than 20µm and each having a higher Dv.99 and/or a higher Dv.50 than the coloured film-forming base component (1) or, when there is more than one such component, than those components taken together, in an amount of from 10 to 99% by weight of the total film-forming components, and, if desired,

(b) one or more other uncoloured film-forming components, in an amount of up to 30% by weight of the total film-forming components.

5. A powder coating composition as claimed in any one of claims 1 to 4, wherein there are two or more coloured film-forming base components (1).

6. A powder coating composition in which powder particles are an agglomerate of individual particulate components fused or bonded together into composite particles, wherein the individual particulate components comprise

(1) two or more coloured film-forming base components having a Dv.99 of no more than 30µm, and

(2) one or more uncoloured film-forming components, at least one component (2a) having a Dv.99 of more than 20µm and that is higher than the Dv.99 of

component (1) or, when there is more than one such component, of those components taken together,
the ratio of coloured film-forming base components (1) to uncoloured film-forming component(s) (2) in the composition being in the range of from 1:99 to 30:70 by weight.

5

7. A powder coating composition as claimed in claim 6, wherein the uncoloured film-forming component or components (2a) are present in an amount of at least 10% by weight of the total film-forming components.

10

8. A powder coating composition as claimed in claim 6 or claim 7, wherein there is no more than 30% by weight, calculated on the weight of the total film-forming components, of uncoloured film-forming component(s) having a Dv.99 (i) that is no higher than the Dv.99 of the coloured film-forming component or components taken together, and (ii) that is less than 20µm.

15

9. A powder coating composition as claimed in any one of claims 1 to 8, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components has a Dv.99 of at least 30µm.

20

10. A powder coating composition as claimed in claim 9, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components has a Dv.99 of at least 35µm.

25

11. A powder coating composition as claimed in claim 10, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components has a Dv.99 of at least 40µm.

12. A powder coating composition in which powder particles are an agglomerate of individual particulate components fused or bonded together into composite particles, wherein the individual particulate components comprise

- 5 (1) two or more coloured film-forming base components having a Dv.99 of no more than 30 μ m, and
- (2) one or more uncoloured film-forming components, at least one component (2a) having a Dv.99 of more than 40 μ m,

the ratio of coloured film-forming base components to uncoloured film-forming
10 component(s) in the composition being in the range of from 1:99 to 60:40 by weight.

13. A powder coating composition as claimed in claim 12, wherein the uncoloured film-forming component or components (2a) are present in an amount of at least 10% by weight of the total film-forming components.

15

14. A powder coating composition as claimed in claim 12 or claim 13, wherein there is no more than 30% by weight, calculated on the weight of the total film-forming components, of uncoloured film-forming component(s) having a Dv.99 (i) that is no higher than the Dv.99 of the coloured film-forming component or components
20 taken together, and (ii) that is less than 20 μ m.

15. A powder coating composition as claimed in any one of claims 1 to 14, wherein the ratio of coloured film-forming base components (1) to uncoloured film-forming component(s) (2a) is in the range of from 1:99 to 50:50 by weight.

25

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16. A powder coating composition as claimed in claim 15, wherein the ratio of coloured film-forming base components (1) to uncoloured film-forming component(s) (2a) is in the range of from 1:99 to 40:60 by weight.

5 17. A powder coating composition as claimed in claim 16, wherein the ratio of coloured film-forming base components (1) to uncoloured film-forming component(s) (2a) is in the range of from 1:99 to 30:70 by weight.

10 18. A powder coating composition as claimed in claim 17, wherein the ratio of coloured film-forming base components (1) to uncoloured film-forming component(s) (2a) is in the range of from 5:95 to 30:70.

15 19. A powder coating composition as claimed in any one of claims 1 to 18, wherein the uncoloured film-forming component or components (2) are present in an amount of at least 50% by weight of the total film-forming components.

20 20. A powder coating composition as claimed in claim 19, wherein the uncoloured film-forming component or components (2) are present in an amount of at least 60% by weight of the total film-forming components.

21. A powder coating composition as claimed in claim 20, wherein the uncoloured film-forming component or components (2) are present in an amount of at least 70% by weight of the total film-forming components.

25 22. A powder coating composition as claimed in any one of claims 2 to 21, wherein the uncoloured film-forming component or components (2a) are present in an amount of at least 20% by weight of the total film-forming components.

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23. A powder coating composition as claimed in claim 22, wherein the uncoloured film-forming component or components (2a) are present in an amount of at least 30% by weight of the total film-forming components.

5

24. A powder coating composition as claimed in claim 23, wherein the uncoloured film-forming component or components (2a) are present in an amount of at least 40% by weight of the total film-forming components.

10

25. A powder coating composition as claimed in claim 24, wherein the uncoloured film-forming component or components (2a) are present in an amount of at least 50% by weight of the total film-forming components.

15

26. A powder coating composition as claimed in claim 25, wherein the uncoloured film-forming component or components (2a) are present in an amount of at least 60% by weight of the total film-forming components.

20

27. A powder coating composition as claimed in any one of claims 1 to 26, wherein there is no more than 15% by weight, calculated on the weight of total film-forming components, of uncoloured film-forming component(s) (2b).

25

28. A powder coating composition as claimed in any one of claims 1 to 27, wherein there are two or more compatible coloured film-forming base components (1).

29. A powder coating composition as claimed in any one of claims 1 to 28, wherein the uncoloured film-forming component (2a) or at least one of the uncoloured

film-forming components listed under (2a) and (2b) is compatible with the coloured film-forming base components (1) during film-formation.

30. A powder coating composition as claimed in any one of claims 1 to 29,
5 wherein the uncoloured film-forming component (2a) or at least one of the uncoloured film-forming components listed under (2a) and (2b) is incompatible with the coloured film-forming base component(s) (1) or becomes incompatible therewith during film-formation.

10 31. A powder coating composition as claimed in any one of claims 1 to 30, wherein the coloured film-forming base component(s) (1) contain a polyester.

32. A powder coating composition as claimed in claim 30 and claim 31,
wherein the uncoloured film-forming component (2a) or at least one of the uncoloured
15 film-forming components listed under (2a) and (2b) contains a polyester having a different functionality from the polyester of the coloured film-forming base component(s) (1).

33. A powder coating composition as claimed in claim 31 or claim 32, which
20 includes an uncoloured film-forming component (2a) or (2b) containing a polyester, that component and the coloured film-forming base component(s) (1) containing different curing agents, one of those being an epoxy curing agent or a co-reactable epoxy resin.

34. A powder coating composition as claimed in any one of claims 1 to 33,
25 which includes an uncoloured film-forming component (2a) or (2b) which is itself an agglomerate of an uncoloured film-forming component fused or bonded to form composite particles with a non-film-forming component.

35. A powder coating composition as claimed in any one of claims 1 to 34, wherein component (2a) or (2b) includes a texturing additive.

5 36. A powder coating composition as claimed in claim 34, wherein the agglomerated component (2a) or (2b) includes mica.

37. A powder coating composition as claimed in any one of claims 1 to 36, wherein the uncoloured film-forming component (2a) or at least one of the uncoloured
10 film-forming components (2a) has a Dv.99 of no more than 90µm.

38. A powder coating composition as claimed in any one of claims 1 to 37, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components (2a) has a Dv.90 of at least 14µm.

15 39. A powder coating composition as claimed in claim 38, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components (2a) has a Dv.90 of at least 18µm.

20 40. A powder coating composition as claimed in any one of claims 1 to 39, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components (2a) has a Dv.90 of no more than 75µm.

25 41. A powder coating composition as claimed in any one of claims 1 to 40, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components (2a) has a Dv.99 in the range of from 50 to 65µm.

42. A powder coating composition as claimed in any one of claims 1 to 41, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components (2a) has a Dv.50 in the range of from 5 to 45 μ m.

5

43. A powder coating composition as claimed in claim 42, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components (2a) has a Dv.50 of at least 8 μ m.

10

44. A powder coating composition as claimed in claim 43, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components (2a) has a Dv.99 of at least 40 μ m and a Dv.50 of at least 10 μ m

15

45. A powder coating composition as claimed in claim 43 or claim 44, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components (2a) has a Dv.50 in the range of from 12 to 30 μ m.

20

46. A powder coating composition as claimed in any one of claims 1 to 45, wherein the coloured film-forming base components or at least one of the coloured film-forming base components (1) have a Dv.99 in the range of from 6 to 25 μ m.

25

47. A powder coating composition as claimed in any one of claims 1 to 46, wherein the coloured film-forming base components or at least one of the coloured film-forming base components (1) have a Dv.50 of no more than 18 μ m.

48. A powder coating composition as claimed in claim 47, wherein the coloured film-forming base components or at least one of the coloured film-forming base components (1) have a Dv.50 of no more than 15µm.

5 49. A powder coating composition as claimed in claim 48, wherein the coloured film-forming base components or at least one of the coloured film-forming base components (1) have a Dv.50 in the range of from 2 to 12µm.

10 50. A powder coating composition as claimed in any one of claims 1 to 49, wherein the coloured film-forming base components contain in total from 5 to 70 weight % of pigment, calculated on the total weight of those components.

15 51. A powder coating composition as claimed in any one of claims 1 to 50, wherein the coloured film-forming base components contain in total from 0.5 to 50 weight % of pigment, calculated on the total weight of the composition.

20 52. A powder coating composition as claimed in any one of claims 1 to 51, wherein the individual particulate components of the agglomerate include one or more non-film-forming component (3).

53. A powder coating composition as claimed in claim 52, wherein the individual particulate components of the agglomerate include a texturing agent (3).

25 54. A powder coating composition as claimed in claim 52 or claim 53, wherein the non-film-forming component or components constitute up to 50% by weight of the total particulate components of the composition.

55. A kit comprising the following separate particulate components for agglomerating into powder coating compositions for the preparation of powder coatings in a number of different colours:

- 5
- a plurality of differently coloured film-forming base components, each with a Dv.99 of no more than 30 μ m, and
 - one or more uncoloured film-forming components, each having a higher Dv.99, and/or a higher Dv.50, each Dv.99 being more than 40 μ m.

10 56. A kit as claimed in claim 55, wherein the uncoloured film-forming component or at least one of the uncoloured film-forming components has a Dv.99 of no more than 90 μ m.

15 57. A kit as claimed in claim 55 or claim 56, which includes at least 7 differently coloured film-forming base components.

20 58. A kit as claimed in any one of claims 55 to 57, which includes an uncoloured film-forming component that is compatible during film-formation with the coloured film-forming base components, and an uncoloured film-forming component that is incompatible with the coloured film-forming base components or that becomes incompatible therewith during film-formation.

25 59. A kit comprising the following separate particulate components for the manufacture of agglomerated powder coating compositions for the preparation of powder coatings in a number of different colours:

- at least 7 differently coloured film-forming base components,

- an uncoloured film-forming component that is compatible with the coloured film-forming base components and remains compatible during film-formation and that has a Dv.99 of more than 40µm and no more than 90µm, and
- an uncoloured film-forming component that is incompatible with the coloured film-forming base components or that becomes incompatible therewith during film-formation, and that has a Dv.99 of more than 40µm and no more than 90µm.

60. A kit as claimed in claim 59, which includes means for comminution of the coloured film-forming base components to a powder having Dv.99 of no more than 30µm.

61. A kit as claimed in any one of claims 55 to 60, wherein the different components are as specified in any one of claims 31 to 53.

62. A kit as claimed in any one of claims 55 to 61, which includes at least 3 uncoloured film-forming components.

63. A kit as claimed in any one of claims 55 to 62, which includes means for agglomerating the components to produce a fluidisable powder.

20

64. A process for the preparation of a powder coating composition as claimed in any one of claims 1 to 54, which comprises providing the specified one or more coloured film-forming base components (1) and the one or more specified uncoloured film-forming components (2a) and if desired one or more other components selected from other uncoloured film-forming components (2b) and non-film-forming components (3), in the specified proportions, and mixing and agglomerating the

components into composite particles such that the composition is air-fluidisable and can be applied to a substrate by electrostatic spray.

65. A process for the preparation of a powder coating composition as
5 claimed in claim 1 from a kit comprising a plurality of differently coloured film-forming
base components and one or more uncoloured film-forming components having a
Dv.99 of more than 40µm, which comprises comminuting at least one of the coloured
film-forming base components to provide a powder having a Dv.99 of no more than
30µm, and mixing and agglomerating the comminuted coloured film-forming base
10 component(s) and at least one of the specified uncoloured film-forming components
having a Dv.99 or a Dv.50 higher than the Dv.99 or Dv.50 respectively of the
comminuted coloured film-forming base component or components taken together, in
the proportions specified in claim 1, to form composite particles, such that the
composition is air-fluidisable and can be applied to a substrate by electrostatic spray.

15

66. A powder coating composition when prepared by a process as claimed
in claim 64 or claim 65.

67. A process for forming a coating on a substrate, which comprises
20 applying an agglomerated composition as claimed in any one of claims 1 to 54 or claim
66 to a substrate, and heating the applied composition to form a continuous coating.

68. A substrate coated by a process as claimed in claim 67.

DECLARATION FOR NON-PROVISIONAL PATENT APPLICATION*

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below at 201 et seq. beneath my name.

I believe I am the original, first and sole inventor if only one name is listed at 201 below, or an original, first and joint inventor if plural names are listed at 201 et seq. below, of the subject matter which is claimed and for which a patent is sought on the invention entitled

POWDER COATING COMPOSITIONS

and for which a patent application:

☐ is attached hereto and includes amendment(s) filed on _____ (if applicable)
☒ was filed in the United States as a national stage application on September 7, 2001 as Application No. 09/936,016 (for declaration not accompanying application) with amendment(s) filed on _____ (if applicable)
☒ was filed as PCT international Application No. PCT/GB00/00871 on March 9, 2000 and was amended under PCT Article 19 on March 20, 2001 (if applicable)

I hereby state that I have reviewed and understand the contents of the above identified application, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119(a)-(d) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

EARLIEST FOREIGN APPLICATION(S), IF ANY, FILED PRIOR TO THE FILING DATE OF THE APPLICATION				
APPLICATION NUMBER	COUNTRY	DATE OF FILING (day, month, year)	PRIORITY CLAIMED	
9905522.0	Great Britain	10 March 1999	YES <input checked="" type="checkbox"/>	NO <input type="checkbox"/>
			YES <input type="checkbox"/>	NO <input type="checkbox"/>
			YES <input type="checkbox"/>	NO <input type="checkbox"/>

I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below.

PROVISIONAL APPLICATION NUMBER	FILING DATE

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information known to me which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

NON-PROVISIONAL APPLICATION SERIAL NO.	FILING DATE	STATUS		
		PATENTED	PENDING	ABANDONED

* for use only when the application is assigned to a company, partnership or other organization.

POWER OF ATTORNEY

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of: CORDINER

Application No.: 09/936,016

Group Art Unit: To be assigned

Filed: September 7, 2001

Examiner: To be assigned

For: POWDER COATING COMPOSITIONS

Attorney Docket No.: 156-024-999

**POWER OF ATTORNEY BY ASSIGNEE
AND EXCLUSION OF INVENTOR(S) UNDER 37 C.F.R. 3.71**Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

The undersigned assignee of the entire interest in the above-identified subject application hereby appoints: Berj A. Terzian (Reg. No. 20060), David Weild, III (Reg. No. 21094), Jonathan A. Marshall (Reg. No. 24614), Barry D. Rein (Reg. No. 22411), Stanton T. Lawrence, III (Reg. No. 25736), Charles E. McKenney (Reg. No. 22795), Philip T. Shannon (Reg. No. 24278), Francis E. Morris (Reg. No. 24615), Charles E. Miller (Reg. No. 24576), Gidon D. Stern (Reg. No. 27469), John J. Lauter, Jr. (Reg. No. 27814), Brian M. Poissant (Reg. No. 28462), Brian D. Coggio (Reg. No. 27624), Rory J. Radding (Reg. No. 28749), Stephen J. Harbulak (Reg. No. 29166), Donald J. Goodell (Reg. No. 19766), Thomas E. Friebe (Reg. No. 29258), Laura A. Coruzzi (Reg. No. 30742), Jennifer Gordon (Reg. No. 30753), Geraldine F. Baldwin (Reg. No. 31232), Victor N. Balancia (Reg. No. 31231), Samuel B. Abrams (Reg. No. 30605), Steven I. Wallach (Reg. No. 35402), Marcia H. Sundeen (Reg. No. 30893), Paul J. Zegger (Reg. No. 33821), Edmond R. Bannon (Reg. No. 32110), Bruce J. Barker (Reg. No. 33291), Adriane M. Antler (Reg. No. 32605), Thomas G. Rowan (Reg. No. 34419), James G. Markey (Reg. No. 31636), Thomas D. Kohler (Reg. No. 32797), Scott D. Stimpson (Reg. No. 33607), Gary S. Williams (Reg. No. 31066), Ann L. Gisolfi (Reg. No. 31956), Todd A. Wagner (Reg. No. 35399), Scott B. Familant (Reg. No. 35514), Kelly D. Talcott (Reg. No. 39582), Francis D. Cerrito (Reg. No. 38100), Anthony M. Insogna (Reg. No. 35203), Brian M. Rothery (Reg. No. 35340), Brian D. Siff (Reg. No. 35679), Alan Tenenbaum (Reg. No. 34939), Michael J. Lyons (Reg. No. 37386), Garland T. Stephens (Reg. No. 37242), William

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An assignment of the entire interest in the above-identified subject application:

[] was recorded on _____ at reel/frame / ____.

[X] is submitted herewith for recording.

Please direct all correspondence for this application to customer no. 20582.

ASSIGNEE: International Coatings Limited

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

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